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DENSITIES, VISCOSITIES AND SPEEDS OF SOUND OF BINARY MIXTURES OF N- ETHYLANILINE WITH TOLUENE, AND NITROTOLUENESAT 303.15 K AND 308.15 K

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ABSTRACT

Physic-chemical properties viz., density (ρ), speed of sound (u), and viscosity (η) at temperatures 303.15 K and 308.15 K are measured for the binary mixtures of N-ethylaniline (N-EA) with toluene (T), o-nitrotoluene (o-NT), and m- nitrotoluene (m-NT) and over the entire range of mixture composition. These data have been used to calculate the excess volume (V^E), deviations in isentropic compressibility ($\Delta\kappa_s$), deviation in viscosity ($\Delta\eta$) and excess Gibbs free energy of activation of viscous flow (G^{*E}). These results are fitted to the Redlich-Kister polynomial equation to derive the binary coefficients and standard errors. The experimental and calculated quantities are used to study the nature of mixing behaviours between the mixture components.

KEYWORDS: Excess Volume, Ultrasonic Velocity, Viscosity, Intermolecular Interaction, N-Ethyl Aniline, Nitrotoluenes

INTRODUCTION

Density and viscosity of liquid mixtures are required in most engineering calculations and different analytical applications where fluid flow or mixing is an important factor. Knowledge about the density and viscosity of multi-component mixtures are also important for understanding and rationalizing the behavior of mixed liquids. The studies of excess and deviation properties are of considerable interest in understanding the intermolecular interactions in the multi-component liquid mixtures. Therefore, evaluation and prediction of these properties of solvent mixtures as functions of temperature and composition are of theoretical and practical importance. This paper is part of our research of the thermodynamic properties [1-5] of binary mixtures containing different alcohols, aromatic ketones and aliphatic ketones, which have great importance as non-electrolyte solvents in various industries. In the chemical industry knowledge of the thermodynamics properties of non-electrolyte solutions is essential in the design involving chemical separation, heat transfer, mass transfer and fluid flow. Furthermore, thermodynamic properties of binary mixtures containing components capable of undergoing specific interactions exhibit significant deviations from ideality arising not only from differences in molecular size and shape but also due to structural changes. The characterization of the mixtures through their thermodynamic and transport properties is important from the fundamental viewpoint to understand their mixing behavior (molecular interactions) as well as in practical applications.

The liquids were chosen in the present investigation on the basis of their industrial importance**.** N-ethylaniline is used as an intermediate to manufacture dyes, agrochemicals and in preparation of some organic compounds. Toluene is a non-polar has slightly polar nature due to delocalized electron cloud, which results in the solute-solvent molecular associates. Nitrotoluene is used in the synthesis of intermediates for azodyes, sulfur dyes, rubber chemicals and agriculture chemicals.

Several researchers elucidated in the literature for density, viscosity and ultrasonic studies of binary mixtures of chlorotoluenes and nitrotoluenes with acetophenone[6], dimethylformamide [7], and with dimethysulphoxide [8]. However, in the present study, the effect of introduction of a nitro group and their orientation in toluene molecule that may influence both the sign and magnitude of excess volume and deviation in isentropic compressibility. In an effort to investigate this effect, we have measured density (ρ), speed of sound (u) and viscosity (η) of mixtures of N-ethylaniline with toluene, + o-nitrotoluene, and m-nitrotoluene and those of their binary mixtures over the entire composition range at 303.15 K and 308.15 K. From the above experimental data several excess thermodynamic parameters like excess volume (V^E), deviations in ultrasonic speed of sound (Δu), deviations in isentropic compressibility ($\Delta\kappa_s$), deviation in intermolecular free length (ΔL_f), deviation in acoustic impedance (ΔZ), deviation in viscosity ($\Delta\eta$) and excess Gibbs free energy of activation of viscous flow (G^{*E}) are evaluated and reported. The variations of these properties with composition for all the binary mixtures are studied in terms of molecular interactions between component molecules.

EXPERIMENTAL

The mass fraction purity of all the liquids from Merck, S.D. Fine Chemicals Ltd., India was as follows: N-ethylaniline (99.7%), toluene (99.7%), o-nitrotoluene (99.5%) and m-nitrotoluene (99.5%). Prior to experimental measurements, all liquids were used after double distillation and partially degassed with a vacuum pump under an inert atmosphere. The purity of these solvents was ascertained by comparing the measured density and viscosity of the pure components with available literature values [6,9-11] as shown in Table 1. The purities of the samples were further confirmed by GLC single sharp peaks. Before use, the chemicals were stored over 0.4 nm molecular sieves for approximately 72 hrs to remove water and were then degassed.

The binary mixtures of N-ethylaniline with toluene, o-nitrotoluene and m-nitrotoluene were prepared in glass bottles with air - tight stoppers, and adequate precautions were taken to minimize losses through evaporation. The weighing of solutions was made using Afcoset ER-120A electronic balance with a precision of ±0.1 mg. The uncertainty in solution composition expressed in mole fraction was found to be less than 1×10^{-4}. After mixing the sample, the bubble-free homogeneous sample was transferred into the U-tube of the densimeter using a syringe. The density measurements were performed with a Rudolph Research Analytical digital densimeter (DDH-2911 Model), equipped with a built-in solid-state thermostat and a resident program with accuracy of temperature of 303.15 K ± 0.03 K. The estimated accuracy in the density measurement is $\pm\ 2\times10^{-5}$ $gm.cm^{-3}$. Proper calibration at each temperature was achieved with doubly distilled, deionized water and with air as standards. A multi – frequency ultrasonic interferometer (M-82 Model, Mittal Enterprise, New Delhi, India) operated at 2 MHz was used to measure the ultrasonic velocities of binary liquid mixtures at constant temperature of 303.15 K controlled by a digital constant temperature water bath. The uncertainty in the measurement of ultrasonic speed is ± 0.2%

The viscosities of pure liquids and their mixtures were determined at atmospheric pressure and at temperature 303.15 K and 308.15 K by using an Ubbelohde viscometer, which was calibrated with benzene and doubly distilled water. The Ubbelohde viscometer bulb has a capacity of 15 ml and the capillary tube with a length of about 90 mm with 0.5 mm internal diameter. The viscometer, thoroughly cleaned and perfectly dried, was filled with the sample liquid by fitting the viscometer to about 30^o from the vertical and its limbs were closed with Teflon caps to avoid the evaporation. The viscometer was kept in a transparent walled bath with a thermal stability of ± 0.01 K for about 20 min to obtain thermal equilibrium. An electronic digital stopwatch with an uncertainty ± 0.01 s was used for flow time measurements. The viscosity values of pure liquids and mixtures are calculated using the relation

$$\eta = (at-b/t)\rho \quad (1)$$

where a and b are the characteristic constants of the viscometer, ρ is the density, and t represents the flow time. The uncertainty of viscosity thus estimated was found to be ±0.005 mPa.s.

THEORY AND CALCULATIONS

The experimental values of density (ρ), ultrasonic velocity (u), and viscosity (η) of pure liquids and their mixtures as function of mole fraction of N-ethylaniline at 303.15 K and 308.15 K were used to calculate the parameters such as excess volume (V^E), deviation in ultrasonic velocity (Δu), deviations in isentropic compressibility ($\Delta\kappa_s$), and deviation in viscosity Δη from experimental data using the following expressions:

$$V^E = [x_1M_1 + x_2M_2]/\boldsymbol{\rho} – [x_1M_1/\boldsymbol{\rho}_1+ x_2M_2/\boldsymbol{\rho}_2] \quad (2)$$

$$\Delta u = u – [x_1\, u_1 + x_2\, u_2] \quad (3)$$

$$\Delta\kappa_s = \kappa_s – [x_1\, \kappa_{s\,1} + x_2\, \kappa_{s\,2}] \quad (4)$$

$$\Delta\eta = \eta – [x_1\, \eta_1 + x_2\, \eta_2] \quad (5)$$

In the above equations, M_1, M_2, $\kappa_{s1,}$ $\kappa_{s2,}$ η_1, η_2, u_1, $u_{2,}$ ρ_1, $\rho_{2,}$ ρ, κ_s, η, and u represent molecular weight, isentropic compressibility, viscosity, ultrasonic speed, and density data of component 1, component 2, the and mixture respectively. The data related to excess volume (V^E), deviation in isentropic compressibility ($\Delta\kappa_s$) and deviation in viscosity (Δη) for the binary systems of N-ethylaniline with toluene, o-nitrotoluene, and m- nitrotoluene, are graphically represented in Figures 1, 2 and 3 respectively at 303.15 K and 308.15 K.

RESULTS AND DISCUSSIONS

As can be seen from the number of studies, the experimental V^E values are treated in the following effects: a) physical, b) chemical, and c) structural geometrical contributions. The magnitude and the positive sign of V^E can arise mainly from the factors: (i) Positive contribution indicates that there are no strong specific interactions between components of a mixture. (ii) Predominant intermolecular H-bond stretching of the associated amine molecules in the presence of other substances; (iii) The steric hindrance as unsuitable interstitial accommodation due to different molar volumes and free volumes of unlike molecules. The negative contributions are a consequence of the effects: (i) Strong intermolecular interactions attributed to the charge- transfer complex, dipole–dipole and dipole–induced dipole interactions and H-bonding between unlike molecules finally leading to the more efficient packing in the mixture than in the pure liquids; (ii) The structural effects that arise from the geometrical fitting of one component in to other are due to the difference in molar volumes and free volume of pure components, which causes negative contribution to V^E. A perusal of curves in Figure 1 indicates that the factors which are responsible for positive excess volume were dominant in the binary mixtures of N-ethylaniline with nitrotoluenes. On the other hand, the property is negative in the binary mixtures containing toluene.

An examination of curves in Figure 1 show that V^E data is negative for binary mixture of N-ethylaniline with toluene is characterized by specific interactions of the electron-donor-acceptor type in which toluene behaves as an electron donor [12]. The π-electron density in the derivatives of benzene ring depends on nature of the group that is attached to it and the hetero nuclear interaction between components molecules depends on the net electron density in the ring.

The V^E data for the mixtures of N-ethylaniline with o-nitrotoluene and m-nitrotoluene depicted in Figure 1 may be

explained if it is assumed that the N-ethylaniline and nitrotoluene mixtures are characterized by interactions between the π-electron cloud of benzene ring with the delocalized π-electron cloud over the nitrogen and oxygen atoms [13] of the nitro group of nitrotoluenes, resulting in the formation of weak P-complex [14]. Since meta substituent are far away as compared to ortho substituent, the polar character decreases, and as expected positive V^E values are increased. Hence, the algebraic excess volume data for the mixtures of N-ethylaniline with nitrotoluenes fall in the order:

o-nitrotoluene < *m*-nitrotoluene

A perusal of curves in Figure 1 indicate that the excess volume data for the mixture N-ethylaniline with toluene is negative causing stronger intermolecular interactions are prevailing in the mixture and weaker interactions are exhibited by nitrotoluenes. This clearly explains that, introduction of nitro group in toluene molecule is influencing sign and magnitude of excess volume to a significant extent.

Deviation in Isentropic Compressibility ($\Delta\kappa_s$)

Generally, excess isentropic compressibility (($\Delta\kappa_s$) depend on several contributions arising from physical, chemical and structural effects [15].The physical contribution consist of weak dipole-dipole interactions (dispersion forces) causing positive ($\Delta\kappa_s$), where as the geometric fitting of molecules of two unequal sizes in to each others, structures resulting in to negative ($\Delta\kappa_s$) values. Chemical contribution include breaking up of the molecular association in the pure liquid resulting positive $\Delta\kappa_s$ values, while specific interactions such as strong dipole-dipole interactions, and charge - transfer complexes among component molecules leading to negative $\Delta\kappa_s$ values.

A perusal of curves in Figure 2 indicate that the deviation in isentropic compressibility ($\Delta\kappa_s$) for all the systems are negative over the entire composition range at 303.15 K and 308.15 K. These may be attributed to the relative strength of effects which influenced the free space between component molecules, described in the literature [16]. The negative $\Delta\kappa_s$ values arise from changes of "free volume" in the real mixtures and presence of lone pair of electors in N-ethylaniline resulting in the formation charge- transfer complexes [17] leading to positive deviation in sound velocity and negative deviation in isentropic compressibility.

The algebraic $\Delta\kappa_s$ values of values for all the binary systems fall in the order:

o-nitrotoluene > m-nitrotoulene

An examination of data in Table 2 deviation in ultrasonic velocities are positive in all the binary systems over the entire range of composition at 303.15 K and 308.15 K. The sign and magnitude of Δu play an important role in describing molecular rearrangement as a result of the molecular interactions between the component molecules in the mixtures. The positive values of Δu in all binary mixture can be explained on the basis of electron donor-acceptor complex formation between unlike molecules in the mixtures.

A perusal of data in Table 2 shows that the values of Δz are negative for N-ethylaniline with o-nitrotoluene, and m-nitrotoluene whereas for the mixture of N-ethylaniline with toluene is positive over all the entire composition ranges at 303.15 K and 308.15 K. Specific acoustic impedance is a quantity depending on the molecular packing of the systems. The positive values of ΔZ in the N-ethylaniline with toluene mixture suggest the presence of a strong specific interactions between components molecules in the mixtures. However the negative values observed in the case of the N-ethylaniline with nitrotoluenes mixture suggest that the rupture of the hydrogen bond of N-ethylaniline molecules.

The speed of sound in a mixture is mainly influenced by its molecular property. As the implest molecular property is the free length between the surfaces of the molecules, it seems interesting to find the variation of the intermolecular free

length with the mole fraction and temperature. A perusal of data in Table 2 shows that the values of deviation in intermolecular free length are negative for all binary mixtures over all the entire composition ranges at 303.15 K and 308.15 K. The negative contributions of the intermolecular free length indicate the presence of a strong intermolecular interaction between unlike molecules. The results for Δu, ΔZ, and ΔL_f support each other.

Deviation in Viscosity(($\Delta\eta$)

According to Fort and Moore [18] deviation in viscosity tends to become more positive as the strength of the interaction increases. The deviation in viscosity variation gives a qualitative estimation of the strength of the intermolecular interactions. The deviation in viscosities [19] may be generally explained by considering the following factors. (i) The difference in size and shape of the component molecules and the loss of dipolar association in pure component may contribute to a decrease in viscosity and (ii) specific interactions between unlike components such as hydrogen bond and charge transfer complexes may cause for increase in viscosity in mixtures than in pure components. The former effect produces negative in excess viscosity and latter effect produces positive deviation in viscosity.

An examination of curves in Figures 3 suggests that the deviation in viscosity for all the systems are positive over the entire composition range at 303.15 K and 308.15 K. The viscosity of a mixture [20,21] depends on the molecular interactions between the components mixture with strong interactions between different molecules show positive viscosity deviations while for mixtures without specific interactions, viscosity deviations are negative.

For the systems N-ethylaniline with toluene and nitrotoluene are positive over the entire composition range, may be ascribed to the closer approach of the dissimilar molecules due to electrostatic interactions, and possibly of electron donor- acceptor complex formation between the components of the mixtures[18].

The excess Gibbs free energy of activation of viscous flow (G^{*E}) is obtained by the following equation:

$$G^{*E} = RT\,[\ln \eta V - (x_1 \ln\eta_1 V_1 + x_2 \ln\eta_2 V_2)] \qquad (6)$$

where V_1, V_2 and V are the molar volumes of the component 1, and component 2, and molar volume of the mixture, respectively. R and T are gas constant and absolute Temperature respectively.

According to Reed et al [23] the positive deviation in G^{*E} may be attributed to specific interactions between unlike molecules, such as hydrogen bonding, dipole-dipole interaction, charge-transfer complex and dispersion forces, which generally are responsible for negative deviations in the property. In the present work, excess Gibbs free energy of activation of viscous flow (G^{*E}) for all binary systems are positive over the entire composition range at 303.15 K and 308.15 K, further supporting the complex formation between the components of the mixture.

Viscosity Interaction Parameters

With a view towards correlations the viscosities of binary liquid mixtures with those of component liquids and interpreting the molecular interactions in these mixtures. Several equations have been put forward from time to time. These are given in the following text:

Grunberg and Nissan [24] have suggested the following logarithmic relation between the viscosity of the binary mixtures and the pure components.

$$\ln \eta = x_1 \ln \eta_1 + x_2 \ln \eta_2 + x_1 x_2 d_{12} \qquad (7)$$

where d_{12} is a constant proportional to the interchange energy. It may be regarded as an approximate measure of the strength of molecular interactions between the mixing components. Fort and Moore [18] reported that for any binary

liquid mixture, a positive value of d_{12} indicates the presence of specific interactions and a negative value of d_{12} indicates the presence of weak interactions between the unlike molecules. In the present work the positive values of d_{12} may be attributed to the specific interactions between unlike molecules..

Katti and Chaudhri [25] proposed the following equation:

$$\ln \eta V = x_1 \ln V_1 \eta_1 + x_2 \ln V_2 \eta_2 + x_1 x_2 W_{vis/}RT \quad (8)$$

where $W_{vis/}RT$ is an interaction term.

Hind et al [26], suggested an equation for the viscosity of binary liquid mixtures as

$$\eta = x_1^2 \eta_1 + x_2^2 \eta_2 + 2x_1x_2 H_{12} \quad (9)$$

where H_{12} is Hind interaction parameter and is attributed to unlike pair interaction.

The experimental and theoretical values of viscosity of the liquid mixtures calculated using Eqs. 7-9 including standard deviation are presented in Table 4. All the empirical relations gave a reasonable fit, but the viscosity values calculated using Grunberg and Nissan, Katti-Chaudhri ,and Hindet al, relation are in good agreement with the experimental values.

The variation of V^E, $\Delta\kappa_{s,}$ $\Delta\eta$ and Δu with mole fraction were fitted to the Redlich-Kister equation [27] of the type,

$$Y^E=x_1x_2[a_0+a_1(x_1-x_2) + a_2(x_1-x_2)^2] \quad (10)$$

where Y^E is V^E or $\Delta\kappa_s$ or $\Delta\eta$. The values of $a_{0,}$ a_1 and a_2 are the coefficients of the polynomial equation and the corresponding standard deviations, σ obtained by the method of least – squares with equal weights assigned to each point are calculated. The standard deviation (σ) are defined as:

$$\sigma (Y^E) = [\sum(Y^E_{obs} - Y^E_{cal})^2/(n-m)]^{1/2} \quad (11)$$

where n is the total number of experimental points and m is the number of coefficients. The values of $a_{0,}$ a_1 and a_2 are the coefficients is determined by a multiple-regression analysis on the least square method and summarized along with the standard deviations between the experimental and fitted values are presented in Table 5.

CONCLUSIONS

In this paper, the densities, viscosities and speed of sound at 303.15 K and 308.15 K over the entire range of composition of N-ethylaniline with toluene, and nitrotoluenes have been measured. These measured physical property data, excess molar volumes, deviation in viscosity , deviation in ultrasonic speeds and deviations in isentropic compressibility have been calculated and correlated by a Redlich–Kister type polynomial equation to derive the coefficients and standard deviation. The sign and magnitude of these quantities have been discussed in terms of electron-transfer complexes, H-bonding, and dipole-dipole interactions between the mixing components.

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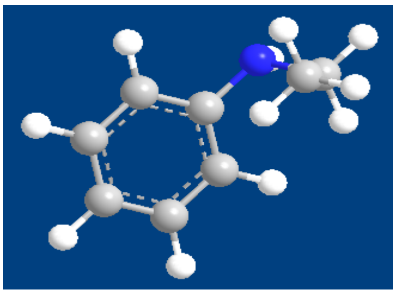
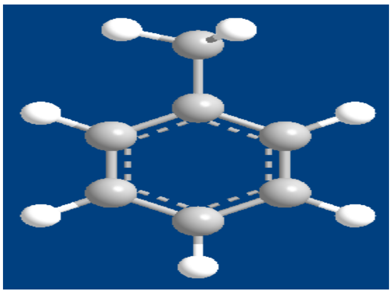
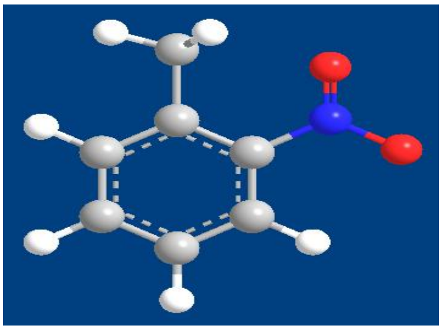
APPENDICES



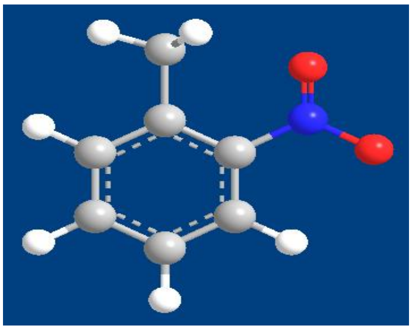
N-Ethylaniline



Toulene



M-Nitrotoulene



O-Nitrotoulene

Figure 1

Table 1: Comparison of Experimental and Literature Values of Density (ρ), Viscosity (η) for Pure Components at 303.15 K

Components	Density (ρ / gm.cm^{-3})		Viscosity (η / mPa.s)	
	Experimental	Literature	Experimental	Literature
N-ethylaniline	0.95274	0.95269[9]	1.742	1.741[9]
Toluene	0.85765	0.85764[10]	0.524	0.525[11]
o-nitrotoluene	1.15325	1.15321 [6]	1.909.	1.911[6]
m- nitrotoluene	1.14764	1.14766 [6]	1.773	1.777[6]

Table 2: Coefficients of Redlich – Kister Equation and Standard Deviation σ Values

Binary Mixtures	Functions	a_0	a_1	a_2	σ
303.15 K					
N-ethylaniline + toluene	V^E / cm^3.mol^{-1}	-1.036	0.082	-0.730	0.001
	Δu / m.sec^{-1}	73.67	4.647	65.66	0.289
	$\Delta\kappa_s$ / TPa^{-1}	-13.98	3.405	-6.213	0.031
	$\Delta\eta$ / mPa.sec	0.837	-0.012	-0.014	0.001
N-ethylaniline+ o-nitrotoluene	V^E / cm^3.mol^{-1}	0.979	0.095	0.239	0.001
	Δu / m.sec^{-1}	99.30	-20.08	-48.38	0.552
	$\Delta\kappa_s$ / TPa^{-1}	-18.29	-4.758	0.577	0.063
	$\Delta\eta$ / mPa.sec	0.117	0.009	-0.052	0.001
N-ethylaniline+ m-nitrotoluene	V^E / cm^3.mol^{-1}	1.196	0.103	0.549	0.001
	Δu / m.sec^{-1}	57.50	3.326	-5.811	0.243
	$\Delta\kappa_s$ / TPa^{-1}	-16.42	-4.395	-1.093	0.079
	$\Delta\eta$ / mPa.sec	0.107	0.006	-0.065	0.001
308.15 K					
N-ethylaniline + toluene	V^E / cm^3.mol^{-1}	-1.067	0.074	-0.934	0.001
	Δu / m.sec^{-1}	70.66	-2.045	44.90	0.162
	$\Delta\kappa_s$ / TPa^{-1}	-14.98	4.110	-5.396	0.044
	$\Delta\eta$ / mPa.sec	0.726	-0.001	-0.288	0.001
N-ethylaniline+ o-nitrotoluene	V^E / cm^3.mol^{-1}	0.879	0.042	0.016	0.002
	Δu / m.sec^{-1}	120.8	-81.55	36.09	0.864
	$\Delta\kappa_s$ / TPa^{-1}	-19.95	-4.194	-0.603	0.039
	$\Delta\eta$ / mPa.sec	0.050	0.004	-0.030	0.001
N-ethylaniline+ m-nitrotoluene	V^E / cm^3.mol^{-1}	1.086	0.129	0.397	0.002
	Δu / m.sec^{-1}	117.78	-36.22	-13.66	1.273
	$\Delta\kappa_s$ / TPa^{-1}	-19.14	-4.718	-0.826	0.043
	$\Delta\eta$ / mPa.sec	0.042	0.002	-0.025	0.001

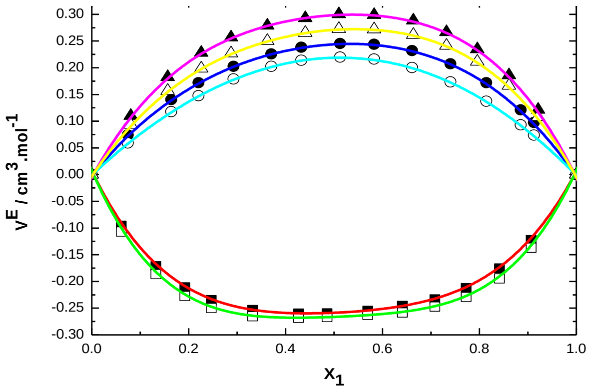


Figure 1: Mole Fraction of N-Ethylaniline (x_1)

Figure 1 Variation of excess molar volume (V^E) with mole fraction (x_1) of N-ethylaniline in the binary liquid mixtures of N-ethylaniline with toluene (■),o - nitrotoluene (●), m- chlorotoluene(▲), respectively at 303.15 K and for N-ethylaniline with toluene (□), o- nitrotouene (O), m- chlorotoluene (Δ) respectively at 308.15 K

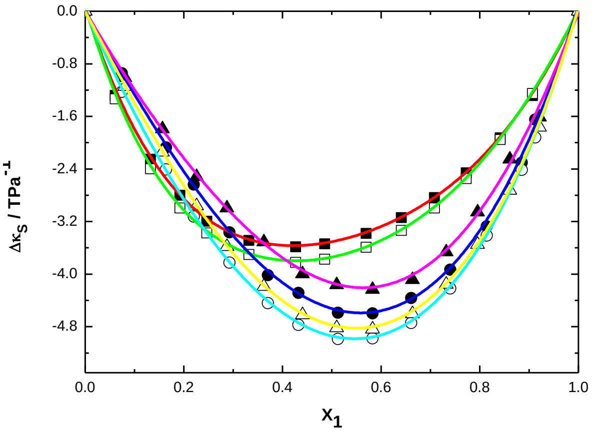


Figure 2: Mole Fraction of N-Ethylaniline (x_1)

Figure 2 Deviation in isentropic compressibility ($\Delta\kappa_s$) with mole fraction (x_1) of N-ethylaniline in the binary liquid mixtures of N-ethylaniline with toluene (■), o- nitrotoluene (●), m- chlorotoluene(▲), respectively at 303.15 K and for N-ethylaniline with toluene (□), o- nitrotoluene (O), and m- chlorotoluene (Δ) respectively at 308.15 K

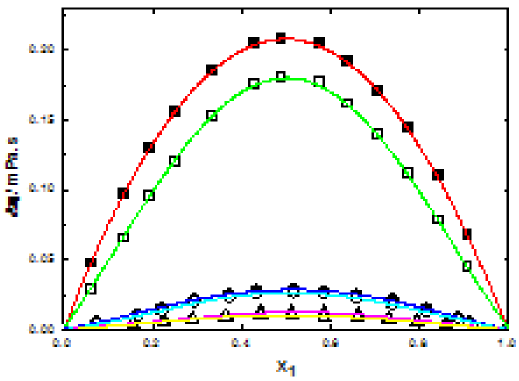


Figure 3: Mole Fraction of N-Ethylaniline (x_1)

Figure 3 Deviation in viscosity $\Delta\eta$ with mole fraction (x_1) of N-ethylaniline in the binary liquid mixtures of N-ethylaniline with toluene (■), o- nitrotoluene (●),m- chlorotoluene(▲), respectively at 303.15 K and for N-ethylaniline with toluene (□), o- nitrotoluene (O), m- chlorotoluene (Δ) respectively at 308.15 K